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**GB**

NATIONAL STANDARD OF THE  
PEOPLE'S REPUBLIC OF CHINA

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**GB/T 13083-2018**

Replacing GB/T 13083-2002

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**Determination of Fluorine in Feeds -  
Ion Selective Electrode Method**

饲料中氟的测定 离子选择性电极法

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Standardization Administration of PRC.**

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## Foreword

This Standard was drafted as per the rules specified in GB/T 1.1-2009.

This Standard replaced GB/T 13083-2002 *Determination of Fluorine in Feed – Ion Selective Electrode Method*; compared with GB/T 13083-2002, this Standard has the major technical changes as follows besides the editorial modifications:

- Expand the scope of the standard “this Standard is applicable to feed, feed ingredients, phosphate and mixed feed additive with aluminosilicate as carrier”;
- Modify the detection limit of the method;
- Modify the description of the principle;
- Modify the measurement range of acidity meter and fluoride ion selective electrode;
- Increase the preparation method of stone powder sample of feed ingredients;
- Increase the preparation method of mixed feed additive sample with aluminosilicate as carrier;
- The result shall be expressed from 0.1mg/kg to 1mg/kg.

This Standard was proposed by and under the jurisdiction of National Technical Committee for Standardization of Feed Industry (SAC/TC 76).

Drafting organization of this Standard: Zhejiang Province Institute of Veterinary Drug and Feedstuff.

Chief drafting staffs of this Standard: Ren Yuqin, Huang Juan, Zhang Zhijian, Zhu Congying, Yuan Lu, Zhang Xiaoli, Qiu Chengjun, and Shang Xiaojin.

The historical editions replaced by this Standard are as follows:

- GB/T 13083-1991, GB/T 13083-2002.

# Determination of Fluorine in Feeds - Ion Selective Electrode Method

## 1 Scope

This Standard specifies using ion selective electrode method to determine the fluorine in the feeds.

This Standard is applicable to the feed, feed ingredients, phosphate and mixed feed additive with aluminosilicate as carrier.

The detection limit of this method is 3mg/kg (take 1g of sample, make constant volume to 50mL).

## 2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use – Specification and Test Methods

GB/T 14699.1 Feeding Stuffs – Sampling

GB/T 20195 Animal Feeding Stuffs – Preparation of Test Samples

## 3 Principle

The sample is extracted by hydrochloric acid solution; use total ionic strength buffer solution to adjust the pH to be 5~6; eliminate the acidity and the ion interference such as  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ , and  $\text{SiO}_3^{2-}$  that can form complexes with fluoride ion; then use ion meter to measure the potential difference between the fluoride ion selective electrode and the saturated calomel electrode; such potential difference is linear with the logarithm of the fluoride ion activity (concentration) in the solution; use the linear equation obtained by the potential difference measured by the fluorine standard series with known concentration to find the fluoride ion concentration corresponding to the potential difference of the unknown sample solution; calculate the fluorine content in the sample.

## 5 Apparatus

- 5.1** Analytical balance: sensitivity of 0.0001g.
- 5.2** Ion meter: measuring range of 0.0mV~±1800mV; or its equivalent pH meter or potentiometer.
- 5.3** Fluoride ion electrode: measuring range of  $10^{-1}$  mol/L ~  $10^{-6}$  mol/L; pF-1 type or its equivalent composite electrode.
- 5.4** Reference electrode: saturated calomel electrode or its equivalent electrode.
- 5.5** Electrothermal constant-temperature drying oven.
- 5.6** High temperature furnace.
- 5.7** Magnetic stirrer.
- 5.8** Ultrasonic extractor.
- 5.9** Nickel crucible or platinum crucible.

## 6 Sample Preparation

Take representative sample according to the provisions of GB/T 14699.1; take about 250g of reduced sample by quadruple method; prepare the sample as per the provision of GB/T 20195; pulverize and pass through the analytical sieve with pore size of 0.425mm; mix evenly; place into the grinding bottle for later-use.

## 7 Test Procedures

### 7.1 Preparation of fluorine standard series

Accurately pipette 0.5mL, 1.00mL, 2.00mL, 5.00mL, 10.00mL of fluorine standard working solution II [4.7c)] and 2.00mL, 5.00mL of fluorine standard working solution I [4.7b)]; separately place them into 50mL volumetric flasks; separately add 5.0mL of hydrochloric acid solution (4.5), 25mL of total ionic strength buffer solution (4.4); add water to the scale; mix evenly. The concentrations of the above standard series are 0.1µg/mL, 0.2µg/mL, 0.4µg/mL, 1.0µg/mL, 2.0µg/mL, 4.0µg/mL, 10.0µg/mL.

### 7.2 Preparation of sample solution

**7.2.1 Preparation of feeds and feed ingredients (except for the three types listed in 7.2.2, 7.2.3 and 7.2.4) sample solutions**

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